

Tribological Challenges in MEMS and Their Mitigation Via Vapor Phase Lubrication

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ABSTRACT

MicroElectroMechanical Systems (MEMS) have become commercially successful in a number of niche applications. However, commercial success has only been possible where design, operating conditions, and materials result in devices that are not very sensitive to tribological effects. The use of MEMS in defense and national security applications will typically involve more challenging environments, with higher reliability and more complex functionality than required of commercial applications. This in turn will necessitate solutions to the challenges that have plagued MEMS since their inception – namely, adhesion, friction and wear. Adhesion during fabrication and immediately post-release has largely been resolved using hydrophobic coatings, but these coatings are not mechanically durable and do not inhibit surface degradation during extended operation.

Tribological challenges in MEMS and approaches to mitigate the effects of adhesion, friction and wear are discussed. A new concept for lubrication of silicon MEMS using gas phase species is introduced. This “vapor phase lubrication” process has resulted in remarkable operating life of devices that rely on mechanical contact. VPL is also an effective lubrication approach for materials other than silicon, where traditional lubrication approaches are not feasible. The current status and remaining challenges for maturation of VPL are highlighted.

Keywords: wear, lubrication, MEMS, friction, vapor, alcohol

1. INTRODUCTION

1.1 Commercial applications

There are a number of commercially-successful MicroElectroMechanical Systems (MEMS) applications, including inkjet printer heads, pressure transducers, micromirror display devices, accelerometers and gyroscopes. The largest volume application is undoubtedly the MEMS accelerometer.¹ These devices have been used in automobile airbag deployment systems for more than 10 years. These devices employ a sensing mass that is suspended over the substrate using microfabricated springs. Electrostatic actuators are used to keep the sensing mass at the neutral position in response to outside accelerations. The voltage required on the actuators to maintain the position of the sensing mass is related to the magnitude of the acceleration that the device is exposed to. Compared to the previous technologies for airbag deployment, MEMS accelerometers are far cheaper than previous technology enabling device redundancy, and include self-test functions and low power operation. A key contributor to the success of these devices is that in normal operation, no contact between surfaces takes place.

Micromirror display devices gained popularity in the late 1990's for use in digital projectors and televisions.² In these applications, light is reflected off of an array of a few hundred thousand to a million individually-addressable mirrors. The mirrors move between two discrete positions corresponding to the pixel being off or on, at frequencies of 10's of kHz. The intensity of the light can be controlled by how long the pixel is in the “on” position, and color is controlled by synchronizing the “on” states with the position of a rapidly spinning color wheel. Millions of colors and intensities can be generated. In more expensive theater displays, a separate micromirror array is used for each of several primary colors, and the resulting pixels are combined on the display screen to generate the desired color and brightness. While this display technology is still used in niche applications, the development of large scale, low cost liquid crystal displays has prevented the micromirror technology from capturing a majority of the market share in consumer televisions.

Another display technology being explored for cellular telephones uses a MEMS membrane to cause interference modulation of the reflected light. By controlling the gap between the membrane and a reflective backing, colors are

generated by optical interference. The result is a very low power display device, and this technology represents significant reductions in battery power required for display compared to current liquid crystal displays.

The next device poised to capture a major market share is the MEMS gyroscope. There are numerous clever designs and sensing methods for determining angular rates in a MEMS device, including capacitive detection of proof mass movement, or exploiting the action of the Coriolis force on resonating rings.³ A MEMS gyroscope has been used in game controllers for several years to translate the motion of a control device to inputs for the game. Vibratory MEMS gyroscopes are also under development for automotive applications, for example in traction control systems.⁴ A major new market for these devices is in smart phones, where they are used for gaming and other operations associated with applications on the phone, such as orienting the display or shuffling a playlist. The popularity of smart phone technology is leading to increased demand for MEMS gyroscopes.

No discussion of potential MEMS applications is complete without the mention of electrical switches. The motivators for the use of electrical switches based on micromachines are high isolation in the off state and low power operation. Most microelectronic switches use some power even in the off state, and can allow leakage of current through the switch. The two types of MEMS switches are capacitive and ohmic. The capacitive switches use a movable element to couple radio frequency energy from a signal line to ground or another signal line, based on the proximity of the movable element to these lines and capacitive coupling of energy across a thin dielectric. These switches are only effective for AC signals. Ohmic MEMS switches rely on direct contact between electrically conductive elements, and can work from DC to high frequency. In both cases, challenges with switches not operating as intended have slowed the development of products based on MEMS switches. With increased operating cycles, the switches either become stuck open, stuck closed, or exhibit increasing contact resistance that lead to unacceptable signal losses.

Examination of the MEMS devices that have been commercially successful reveals that they involve surfaces that do not touch, or where surfaces touch but the amount of sliding is minimal. For example, in the case of the micromirror arrays the corners of the mirrors touch stops at each angular limit of deflection. At these locations, the estimated sliding distance is less than 50 nm, and the contact forces are kept as low as possible to hold the mirror in this state. Furthermore, when the mirror is switched, a complex voltage pulse delivered to the opposing electrode causes a bending wave in the mirror that helps “snap” it off of the landing pad. Since the human eye is sensitive enough to detect a single non-operating pixel, this combination of design and operating parameters was developed to produce mirrors that will reliably switch many billions of times.

The major factors limiting applications of MEMS that involve contacting and sliding surfaces are adhesion, friction and wear.⁵ Solving these problems would increase the design space, and enable more complex device functions relying on surface interactions. One example is shown in Figure 1, which is part of a mechanically discriminating microswitch. Electrostatic actuators (partially shown) drive a small output gear, and this rotary motion is translated through a gear train to a large output gear. The output gear can have several layers, with various gear teeth intentionally omitted. When engaged with a mating gear of the same kind, a precise sequence of moves would be necessary to rotate both gears into a desired position, for example to allow passage of a laser beam through an aperture in the gear.

1.2 Defense applications

Defense applications tend to drive technology developments, and these applications will typically involve more challenging environments with more complex functionality compared to commercial uses. Significant increases in capability also tend to come with lower margins of safety and the requirement to accept lower reliability at the single device level when a technology is new. Fortunately, their small size and batch fabrication ability may allow gains in reliability of MEMS through redundant functionality. In spite of the challenges created by relying on contacting surfaces in design, some attempts to use MEMS for defense applications have been made. One example is a signal discriminating device using metal gears made with MEMS technology.⁶ The authors report “strong” reliability, although giving no mention of lubrication, wear, or number of times the device can operate. Deeds and coworkers⁷ evaluated packaged MEMS structures and diagnostic devices for a defense application and found that moisture ingress leading to adhesion failures were a principal source of reliability problems. Mason and coworkers⁸ identified adhesion and wear as common causes of failure of MEMS for military applications, and have begun to develop a standardized reliability assessment method for MEMS of interest in Department of Defense applications.

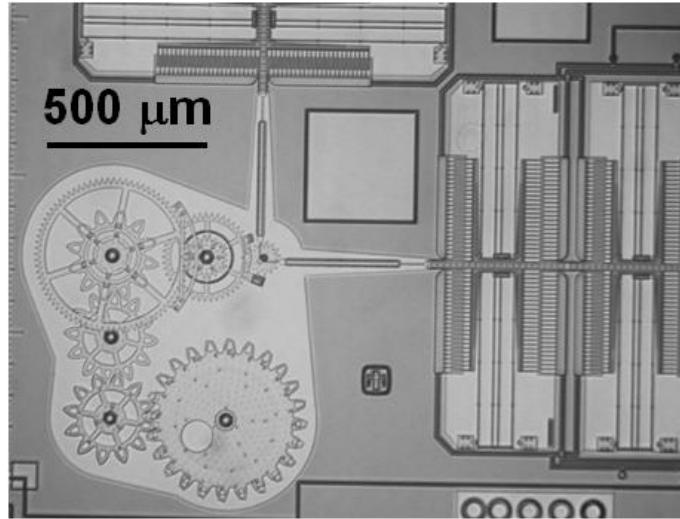


Figure 1. Half of a prototype discriminating microswitch. Two of the large multilayered output gears mated together, with strategically missing gear teeth on different layers, would require a precise sequence of actuations on two separate drive systems in order to move the gear to a desired state. Device courtesy of Marc Polosky, Sandia National Laboratories.

2. TRIBOLOGICAL CHALLENGES

2.1 Adhesion

Since the inception of MEMS, designers have encountered problems due to surface interaction forces that we ignore in most macroscopic applications. Forces due to surface tension in liquid capillaries, electrostatic charge transfer, and van der Waals interactions can be on the order of the actuation forces used to move structures in MEMS, and if not carefully managed will prevent devices from operating as intended.

Several solutions have been developed to reduce MEMS failures due to adhesion. Most of these have addressed adhesion during the manufacturing phase, or “in-process” adhesion. One approach is to reduce the total real surface area in contact. It is well known that two surfaces in contact over an apparent area A will experience true contact over only a small fraction of this area, much less than $0.01*A$, due to the effects of surface roughness. Intentionally roughening a surface can reduce the real contact area, but increases the contact pressure at the remaining contact locations as these must now carry the applied force between the bodies. If the resultant pressures exceed the fracture stress of the material, wear particle generation will result. Polycrystalline silicon MEMS surfaces can be quite easily roughened by intentionally oxidizing the silicon structural layer before the sacrificial oxide is deposited. Oxidation proceeds more rapidly at grain boundaries than away from them, and hence when the sacrificial oxide is etched the oxidized silicon at grain boundaries is also removed, leaving a rougher surface. A large penalty is paid in reduced fracture strength of the silicon structural layers, however, as the oxide etched from grain boundaries creates larger pre-existing crack-like flaws where stress concentrations will lead to fracture.⁹

The common method to mitigate in-process adhesion is with the use of hydrophobic coatings.¹⁰ Since most in-process adhesion is associated with the formation of capillary films between movable structures during device release, hydrophobic coatings are very effective in allowing the manufacture of very complex devices with compliant structures. After etching of the sacrificial layer, devices are typically rinsed in DI water. The water is replaced with a hydrocarbon solvent through a series of mutually miscible solvent replacement steps while keeping the device submerged in fluid at all times. At the final step, the device is left submerged in a solvent for the coating molecules. For instance, silicon devices etched in buffered HF and rinsed in water are transferred to isopropanol, and then to iso-octane. The latter is a solvent for perfluorodecyltrichlorosilane, (FDTs) which is added to iso-octane at about 1 millimolar concentration. The FDTs molecule reacts with –OH sites on the silicon surface to produce a siloxane bond with the silicon, and the molecule is attached 1 monolayer thick. The opposite end of the molecule is terminated in –CF₃, so it is very low energy

and hydrophobic. The devices are then transferred back through the solvent steps to water. When removed from water, the hydrophobic surfaces prevent development of attractive capillary forces to pull compliant structures into contact. There have been many different molecules investigated for use as hydrophobic coatings, with various temperature limits, packing densities, and attachment chemistries. Some are even deposited from the vapor phase¹¹ after releasing the devices and extracting the solvent in supercritical CO₂, to avoid the formation of liquid menisci.

While hydrophobic coatings have enabled the fabrication of very complex devices with compliant structures, they do have some limitations. When heated in ambient air, water vapor can reach the surface at defect sites in the monolayer, which will always exist since they are deposited on rough surfaces where the density of potential bond sites is greater than the areal density of chemisorbed molecules.¹² At these locations, the water facilitates breaking of the siloxane bond and unzips the molecule from the surface. This presents a problem in packaging operations where the die must be heated to over 150°C to degas prior to attaching a hermetic lid. Many lid sealing techniques require heating the device to several hundred degrees C. Hydrophobic coatings degrade rapidly under these conditions.¹³ This difficulty can be avoided by packaging in inert gas or vacuum. The coating molecules will eventually begin to decompose, but at temperatures above most packaging and lid sealing process temperatures.¹⁴

2.2 Friction and wear

An even greater problem with hydrophobic coatings than temperature or environment limits is that they are not mechanically robust. Hook and coworkers¹⁵ used a MEMS device treated with a hydrophobic coating to examine the effects of repeated contact on adhesion in a nitrogen atmosphere. Changes in adhesion force were monitored by noting the voltage corresponding to the separation of two contacting surfaces that were driven into contact at a known force. Figure 2 shows that even in the absence of sliding, a dramatic increase in adhesion force was detected after about 100,000 contacts. Coating degradation occurs more rapidly during sliding, and the resulting surfaces with their hydrophobic coatings removed are subject to failure due to adhesion during operation. This type of adhesive failure is known as “in-use” adhesion.

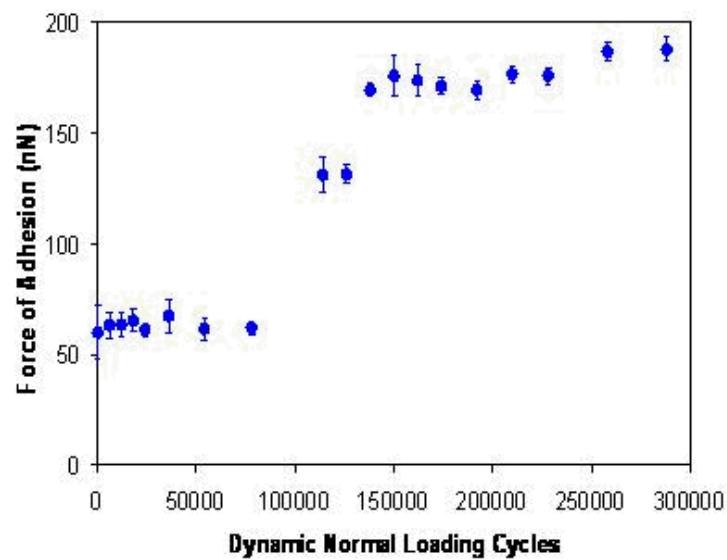


Figure 2. Measurements of adhesion force on a tribology diagnostic device during normal contact in vacuum. After approximately 100,000 normal contacts (without sliding), the adhesion force increases by a factor of three due to damage to the hydrophobic coating. After reference 15.

The loss of hydrophobic coatings makes devices susceptible to in-use adhesion, but can also create unacceptably high friction forces such that available actuation forces are insufficient to move the device. This type of failure was common in devices with electrostatic actuators due to limits on capacitive force produced by the actuators, in the range of a few μ N. Thermal actuators provide much more force, in the range of mN, but are much slower due to the time required to

conduct heat. When sufficient actuation force can be provided to overcome the friction force even after wear of the hydrophobic coatings, devices can be made to move. In this case, however, wear particle generation is likely. MEMS devices that depend on precise positioning of structures, or where particulate contamination of optical surfaces is a concern, cannot tolerate moving surfaces that create particles.

Solid lubricant coatings for MEMS have been investigated, including MoS_2 , WS_2 and diamond-like carbon (DLC) coatings. Deposition methods used for macroscopic parts of complex shape are generally not applicable to fully assembled micromachined devices. Directional processes such as sputtering cannot create uniform lubricant coatings in narrow gaps and at sliding interfaces that are deeply buried in a structure. Plasma assisted chemical vapor deposition (PACVD) used to deposit conformal DLC coatings also cannot penetrate into the deeply buried interfaces that characterize surface micromachined devices. These surfaces are easily reached during the hydrophobic coating processes discussed previously, since one reactant is the surface itself, and the reaction proceeds by diffusion of molecular species to active sites on the surface. A similar coating process has recently been employed to form solid lubricant coatings on MEMS surfaces by Atomic Layer Deposition (ALD).¹⁶ While able to create remarkably uniform coatings at deeply buried interfaces, the reactants needed to form WS_2 coatings cause etching of polycrystalline silicon. This problem can be resolved by first depositing an insulating layer of Al_2O_3 using the same approach, and then depositing WS_2 on top. Unfortunately, the WS_2 is a semiconductor, and while extreme uniformity is advantageous from the perspective of coating deeply buried interfaces, it also creates current leakage paths on the structure so that electrical actuation or sensing signals are shorted to ground.

It would seem that many of the challenges associated with friction and wear in MEMS could be solved by simply fabricating the structural elements from materials other than silicon that are naturally hydrophobic, self-lubricating or extremely resistant to wear.¹⁷ Several such materials have been investigated, including SiC and nanocrystalline diamond. These do not present a simple solution, however, due to difficulties patterning and etching these materials, finding suitable sacrificial layers and selective etchants for those, and controlling residual stress in the structural films as well as the sacrificial layers. These problems have been solved in silicon with silicon dioxide sacrificial layers since micromachine fabrication builds upon several decades of processing knowledge for silicon developed during semiconductor device manufacturing. A fabrication process has been developed for SiC MEMS,¹⁸⁻¹⁹ but relies upon heated etchants with poor selectivity or slow laser ablation to pattern the films. Nanocrystalline diamond MEMS have been developed as well.²⁰ In this case etching is straightforward using an oxygen plasma, and SiO_2 sacrificial layers can be used. However, growth of successive layers of diamond and SiO_2 to create multilayer structures has not been demonstrated. The residual stress in the films is also problematic. Residual stress reported to be “low” by investigators, and which is indeed low compared to $\sim 1\text{GPa}$ compressive stress in DLC films, is still much larger than the $\sim 1\text{MPa}$ residual stress in polycrystalline silicon. Residual stress and through-thickness gradients cause compliant structures to bend out of plane and fail to engage properly with other structures. Small, stiff structures are less susceptible to the effects of residual stress, and there have been some successes in the development of small resonator structures from nanocrystalline diamond.²¹

Operating MEMS in bulk oil is a potential solution to friction and wear problems. Devices can be operated while submerged in fluids as long as they have sufficient dielectric constant to prevent current leakage during actuation. Silicone oil is one example, and silicon devices operate effectively in this environment. One of the principal advantages of MEMS – low inertia enabling rapid operation – is lost when operating in fluids. Reddyhoff and coworkers²² recently investigated fluid additives to minimize boundary friction effects for MEMS operated in liquids, but viscous shear still results in significantly slower operation in liquid compared to gaseous environments. Liquid lubrication is clearly not an option for resonant structures that require high quality factor for operation.

Solutions to the friction and wear problems encountered in microsystems have been sought since MEMS were first developed.²³ Hydrophobic coatings enabled development and release of very complex structures, but did not resist wear sufficiently to allow long term operation. Solid lubricant films and hard coatings either cannot be deposited on fully assembled devices after release, or produce structural or electronic defects in the materials after deposition. While there are many reports of new MEMS materials to solve these problems, a major application has not been identified to create the necessary market pull to warrant the investment required to mature these processing technologies. A low friction, wear resistant material is necessary, but far from sufficient. It must also have low stress, sacrificial layers, and etching processes for both the structural material and the sacrificial layers.

3. A NEW LUBRICATION PARADIGM

3.1 Vapor phase lubrication of silicon

Vapor phase lubrication (VPL) of silicon began as a fundamental investigation of the effects of alcohol adsorption on friction coefficient in pin-on-disk sliding experiments. Asay and coworkers²⁴⁻²⁵ found that when a SiO₂ ball was slid on an oxidized silicon wafer in dry nitrogen, the expected surface damage and wear debris generation occurred. These results are illustrated in Figure 3, where friction coefficient initially increased with number of sliding cycles in nitrogen, and oscillations in friction were exhibited due to the fracture of the silicon surface, release of particles, and those particles becoming first trapped and then released from the contact region. Although this test was stopped after 250 cycles, continued sliding under these conditions for many more cycles produces a reduction in friction coefficient to about 0.3 as shear occurs within a thick layer of debris. This is accompanied by significant wear of the contacting surfaces. When pentanol vapor was added to the environment at concentrations between 8 and 95% of the saturation pressure, the friction coefficient was below 0.2 and no wear could be detected.

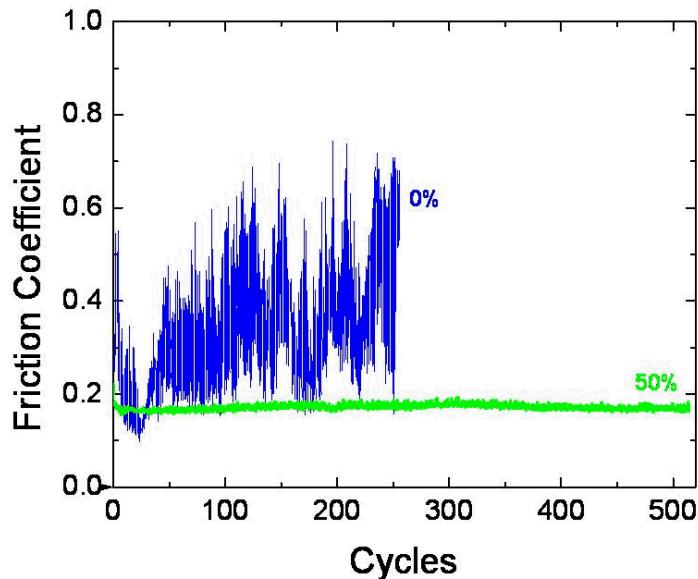


Figure 3. Friction coefficient as a function of sliding cycles for a SiO₂ ball sliding on an oxidized silicon [100] wafer at 98 mN applied load, at 1.5 mm/s. The background atmosphere is dry nitrogen, and the percentages refer to the percentage of the saturation pressure of pentanol present in the environment.

In previous investigations of the effects of adsorbed alcohols on adhesion, Strawhecker and coworkers²⁶ measured the adsorption isotherm of several linear alcohols between 1 and 10 carbon atoms long on oxidized silicon at room temperature. They found that near 10% of the saturation pressure for each alcohol, a full monolayer of the alcohol was adsorbed on the surface. The adsorption isotherms also remained very flat, such that by 90% of the saturation pressure there are only 2-3 monolayers adsorbed on the surface. This is believed to be due to the polar -OH end of the molecule reacting with the surface, thus presenting the low energy -CH₃ group to the vapor which discourages further adsorption. This is exactly the behavior desired for a MEMS lubricant, so that thick fluid layers do not adsorb and lead to liquid-mediated adhesion. Water exhibits the latter behavior, where the adsorbed amount of water increases monotonically with partial pressure so that many monolayers of water are adsorbed at partial pressures below saturation.

Asay and coworkers²⁴ used a surface micromachined (SMM) silicon tribology test device to examine the effectiveness of MEMS lubrication with alcohol. Devices treated with hydrophobic coatings and run in dry nitrogen operated for about 3000 cycles before seizing, when the friction force at the contact location exceeded the available electrostatic actuation force. With alcohol in the nitrogen environment between 15 and 95% of saturation pressure, the device would operate with the identical drive signals for weeks before being stopped by the operator. The longest duration of one of these tests corresponded to more than 10⁸ sliding cycles, with no evidence of wear, particle formation, or change in operating characteristics.

Vapor phase lubrication is not a new idea. In its traditional implementation, hydrocarbon gases are exposed to a heated metal surface such that adsorption and decomposition of the gas takes place leaving a carbon film on the metal part.²⁷ This carbon film reduces metallic contact and serves as a lubricant, and is replaced from the gas phase when the metal surface is exposed through wear of the coating. The new aspect of VPL for silicon MEMS is the lubrication of surfaces at room temperature, allowing the adsorption kinetics and reactivity of the surfaces to provide the thermodynamic driving force for bond passivation rather than heating the surfaces to force a particular type of reaction (decomposition in prior work).

Since the original success with lubricating the MEMS tribology diagnostic device with alcohol vapor, many other complex devices have been tested with alcohol VPL and found to have remarkable increases in operating life compared to hydrophobic coatings alone or no surface treatment. The device in Figure 1 provides one such example. With a vapor-deposited hydrophobic coating, this device operates for about 50,000 cycles in air until seizure occurs. Fresh devices run at 50% of the saturation pressure of pentanol operated for 5×10^8 cycles before the experiment was stopped. No wear or particle formation could be observed upon microscopic examination of the device.

The original work by Asay²⁴⁻²⁵ on VPL of silicon with pentanol included time-of-flight secondary ion mass spectrometry (ToF-SIMS) of sliding tracks created during the pin-on-disk tests. This technique was chosen for analysis since it has the surface sensitivity (<< monolayer) and spatial resolution ($\sim 0.25 \mu\text{m}$) required to perform analysis on MEMS contacting surfaces, and the ability to extract chemical information about surface species through their ion fragmentation spectra. ToF-SIMS revealed species in the areas contacted by the SiO_2 ball rubbing on the flat Si with molecular weights of up to 400 amu. The molecular weight of pentanol is just 88 amu, suggesting that sliding in the presence of pentanol vapor was causing surface reactions leading to polymerization into larger hydrocarbons. Barnette and coworkers²⁸ examined the effects of mechanical contact pressure and concentration of pentanol in the atmosphere, and concluded that reaction product formation was not the primary lubrication mechanism, but rather a byproduct of the reaction of pentanol with the SiO_2 surface in contacted areas. This was an important finding, as it suggests that effective MEMS lubrication can be achieved without reaction product accumulation, which may lead to mechanical interference or damping of motion if present in large quantities.

3.2 VPL for other materials

Success with lubrication of silicon with alcohol vapor has prompted investigation of the lubrication mechanism and effectiveness on other materials used in macroscopic electromechanical mechanisms for defense applications. These mechanisms use parts that are conventionally machined from engineering alloys. Figure 4 shows pin-on-disk friction measurements using clean ceramics and engineering alloys in contact with 440C steel balls as the counterpart. The baseline measurements were made in a dry argon atmosphere at a contact load of 98 mN and sliding speed of 1.5 mm/s, similar to the prior work with silicon. The unlubricated sliding experiments revealed friction coefficients above 0.4 for all material combinations, and oscillations in friction indicative of adhesive wear and particle formation. Pentanol was then added to the atmosphere at 20% of the saturation pressure at room temperature, to assure full monolayer coverage. The figure shows that in the case of Si_3N_4 , 440C stainless steel, and aluminum 6061-T6, steady-state friction remained below 0.2, with no particle generation. The pentanol lubrication was ineffective on aerospace titanium alloy Ti6Al4V, which showed no difference in friction performance with and without pentanol vapor. The different behavior in this material is believed to be due to the acid/base character of the surface oxide compared to that present on the other materials. Since Ti6Al4V is an alpha-beta alloy and can have a range of different surface oxides, additional work is necessary with carefully controlled oxides of titanium to better understand these differences.

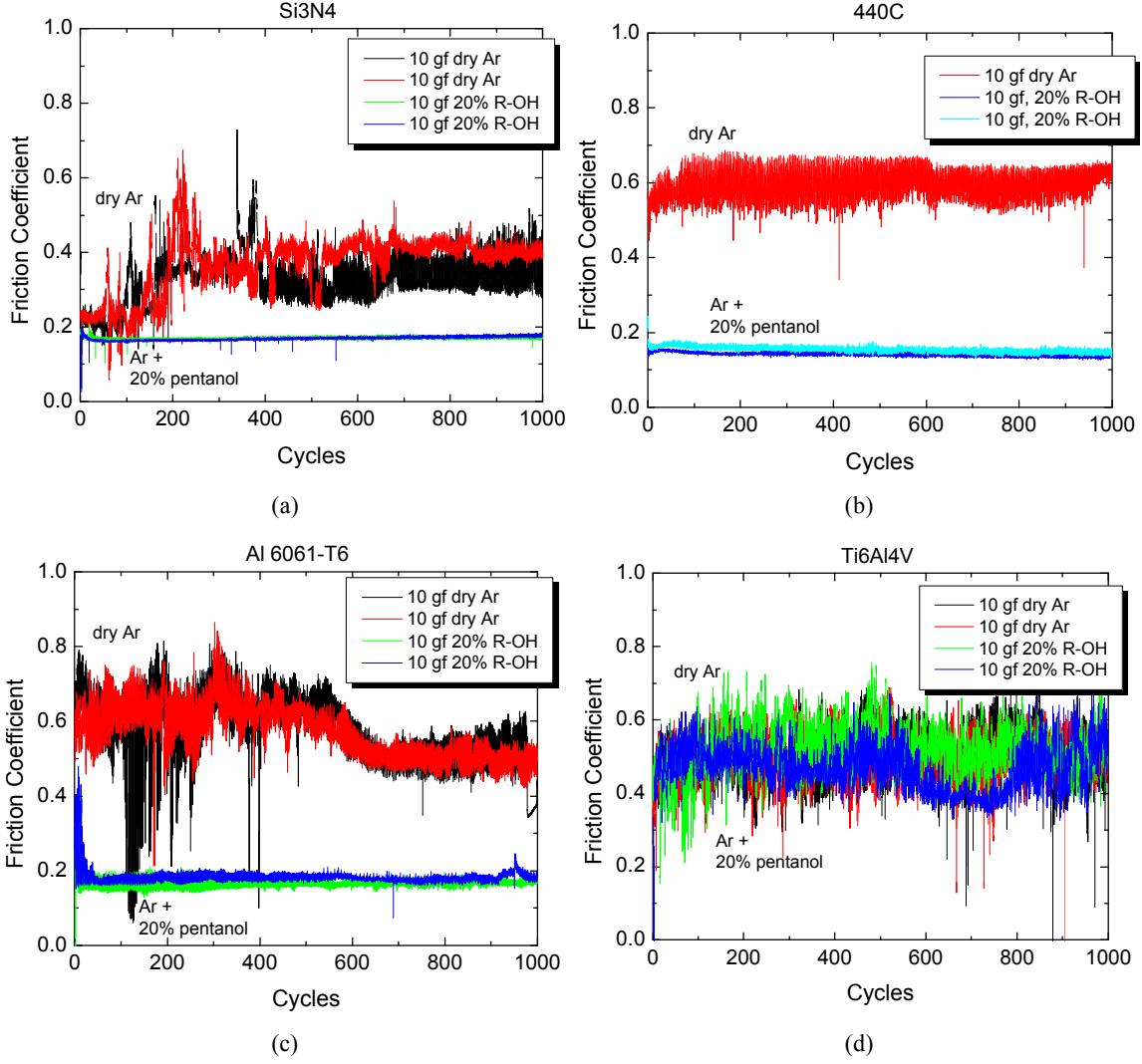


Figure 4. Pin-on-disk friction coefficient measurements for ceramic (a) and engineering alloys (b-d) sliding in dry argon or argon plus 20% of the saturation pressure of pentanol. In all cases but Ti6Al4V, the pentanol vapor reduces friction and wear debris production.

3.3 Remaining challenges in VPL

There are several challenges to mature VPL for use in defense applications. The main challenge is associated with the temperature range over which VPL can be used. A single-component vapor species will have vapor pressure that varies by orders of magnitude over a narrow temperature range, such as several 10's of degrees centigrade. The operating temperature limits are bounded on the low end by the temperature at which a fixed amount of the vapor species in a sealed volume will condense. Condensation will produce liquid films that may interfere with device operation by damping motion or creating capillary films that would increase adhesion, or by interfering with optical surfaces and the transmission of laser light. Operating temperature is bounded on the high end by the need to keep a full monolayer of the vapor species adsorbed on the surface so that broken bonds can be immediately passivated by adsorbed molecules. This situation is illustrated in Figure 5 for pentanol vapor lubrication. For example, a volume filled at 20°C to 10% of the saturation pressure of pentanol will allow operation down to approximately 0°C before condensation of the pentanol will occur, giving just a 20°C operating temperature range. This limitation can be ameliorated to some extent by operating at higher temperature, due to the change in the vapor pressure versus temperature for pentanol. Filling a sealed volume at 100°C with 10% of the saturation pressure will enable operation down to about 55°C before condensation would occur,

giving approximately a 45°C operating temperature window. Investigation of lubrication with other vapors may enable increases in operating temperature ranges by using multi-component vapors, combining materials having very different vapor pressure versus temperature behavior.

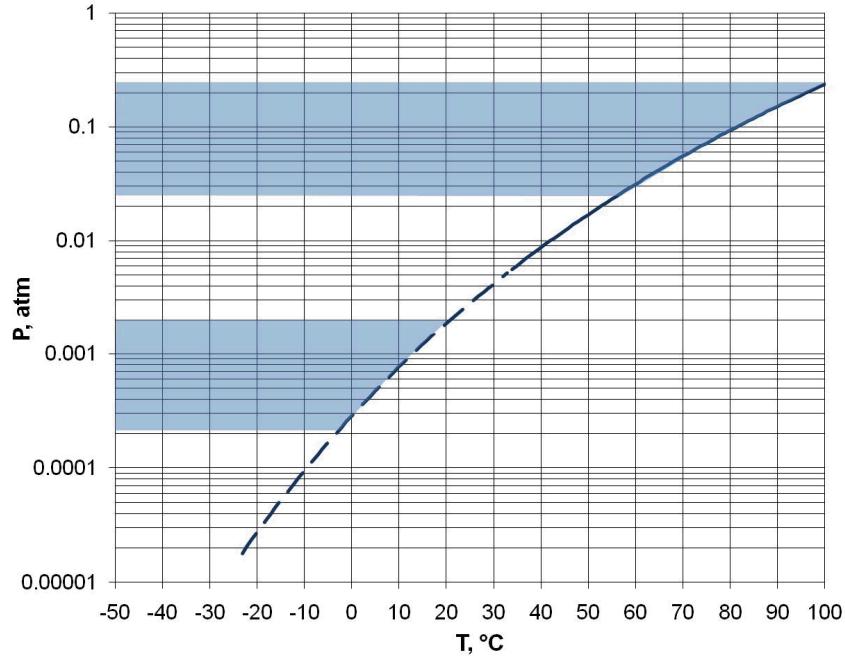


Figure 5. Vapor pressure versus temperature for pentanol based on Antoine equation parameters, after reference 29. The shaded regions correspond to the range of vapor pressures that would lubricate a MEMS device effectively if filled to 10% of the saturation pressure at 20°C and 100°C. Operating at a higher temperature provides a large operating temperature window.

Another requirement for maturation of VPL is development of delivery approaches for the vapor. There are typically enough lubricant molecules in the free volume of an electromechanical device to allow operation for many millions of cycles, so that continuous supply of fresh vapor via a flowing source is not necessary. However, filling a sealed volume with a known quantity of pentanol presents some processing challenges, particularly for MEMS devices. Another approach would be to generate the vapor inside the controlled volume. Recent work suggests that a polymer can be used to generate the desired vapor *in situ*.

4. SUMMARY

The lack of MEMS devices that make use of contacting and rubbing surfaces is a result of the problems associated with lubrication and wear prevention in microsystems. A robust method of lubricating these devices would open up the design space, and enable increased design complexity and associated functionality. Approaches to mitigate in-process adhesion such as hydrophobic coatings do not survive well in mechanical contact, and the use of inherently more wear-resistant materials for fabrication is hampered by the lack of mature micromachining infrastructure for these materials.

Lubrication of silicon, as well as the surfaces of some engineering alloys, using equilibrium adsorption of alcohols from the vapor phase has been demonstrated. In the case of MEMS devices, vapor phase lubrication enables remarkable operating lifetimes without measurable wear, and avoids viscous damping of device motion due to shear of a fluid layer. The adsorbed films behave as a solid lubricant, and are replenished from the vapor phase if removed by contact.

Maturation of VPL for defense applications requires schemes to increase the operating temperature range, and approaches for delivering the desired vapor species inside a controlled volume. Investigation of VPL using other species

may enable increased operating temperature through multi-component vapors. Several delivery approaches are also being investigated, including on-demand generation of the desired vapors through controlled decomposition of polymers.

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REFERENCES

- [1] Eddy, D.S., and Sparks, D.R., "Application of MEMS technology in automotive sensors and actuators," Proc. IEEE 86, 1747-1755 (1998).
- [2] Liao, C.-D. and Tsai, J.-C., "The evolution of MEMS displays," IEEE Trans. Ind. Elect. 4, 1057- 1065 (2009).
- [3] Neul, R., Gómez, U.-M., Kehr, K., Bauer, W., Classen, J., Döring, C., Esch, E., Götz, S., Hauer, J., Kuhlmann, B., Lang, C., Veith, M. and Willig, R., "Micromachined angular rate sensors for automotive applications," IEEE Sensors J. 7, 302-309 (2007).
- [4] Acar, C., Schofield, A.R., Trusov, A.A., Costlow, L.E. and Shkel, A.M., "Environmentally robust MEMS vibratory gyroscopes for automotive applications," IEEE Sensors J. 9, 1895-1906 (2009).
- [5] Romig, A.D. Jr., Dugger, M.T. and McWhorter, P.J., "Materials issues in microelectromechanical devices: science, engineering, manufacturability and reliability," Acta Materialia 51, 5837-5866 (2003).
- [6] Zhang, W., Chen, W., Zhao, X., Li, S., and Jiang, Y., "A novel safety device with metal counter meshing gears discriminator directly driven by axial flux permanent magnet micromotors based on MEMS technology," J. Micromech. Microeng. 15, 1601-1606 (2005).
- [7] Deeds, M., Cochran, K., Swaminathan, R. and Sandborn, P., "Carrier-level packaging and reliability of a MEMS-based safety and arming device," Mat. Res. Soc. Symp. Proc. 682E (2001).
- [8] Mason, R., Ginterta, L., Rippenga, M., Skeltonb, D., Zuninob, J. and Gutmanisc, I., "Guidelines for reliability testing of microelectromechanical systems in military applications," Proc. SPIE 6111, 61110K1-61110K12 (2006).
- [9] Boyce, B.L., Shaw, M.J., Lu, P. and Dugger, M.T., "Stronger silicon for microsystems," Acta Materialia 58, 439-448 (2010).
- [10] Maboudian, R., Ashurst, W.R. and Carraro, C., "Tribological challenges in micromechanical systems," Tribology Letters 12, 95-100 (2002).
- [11] Hankins, M.G., Resnick, P.J., Clews, P.J., Mayer, T.M., Wheeler, D.R., Tanner, D.M., Plass, R.A., "Vapor deposition of amino-functionalized self-assembled monolayers on MEMS," Proc. SPIE 4980, 238-247 (2003).
- [12] Stevens, M.J., "Thoughts on the structure of alkylsilane monolayers," Langmuir 15, 2773-2778 (1999).
- [13] Dugger, M.T., Hohlfelder, R.J. and Peebles, D.E., "Degradation of monolayer lubricants for MEMS," Proc. SPIE 4980, 138-150 (2003).
- [14] Kluth, J.G., Sander, M., Sunga, M.M. and Maboudian, R., "Study of the desorption mechanism of alkylsiloxane self-assembled monolayers through isotopic labeling and high resolution electron energy-loss spectroscopy experiments," J. Vac. Sci. Technol. A16, 932-936 (1998).
- [15] Hook, D.A., Timpe, S.J., Dugger, M.T. and Krim, J., "Tribological degradation of fluorocarbon coated silicon microdevice surfaces in normal and sliding contact," J. Applied Physics 104, 034303,1-034303,6 (2008).
- [16] Scharf, T.W., Prasad, S.V., Dugger, M.T., Kotula, P.G., Goeke, R.S. and Grubbs, R.K., "Growth, structure, and tribological behavior of atomic layer-deposited tungsten disulphide solid lubricant coatings with applications to MEMS," Acta Materialia 54, 4731-4743 (2006).
- [17] Spearing, S.M., "Materials issues in microelectromechanical systems (MEMS)," Acta Materialia 48, 179-196 (2000).
- [18] Mehregany, M., Zorman, C.A., Rajan, N. and Wu, C.H., "Silicon Carbide MEMS for harsh environments," Proc. IEEE 86, 1594-1610 (1998).

- [19] Vendan, M., Molian, P., Bastawros, A. and Anderegg, J., "Ultra-short pulsed laser deposition and patterning of SiC thin films for MEMS fabrication," *Mat. Sci. in Semicond. Proc.* 8, 630-645 (2005).
- [20] Krauss, A.R., Auciello, O., Gruen, D.M., Jayatissa, A., Suman, A., Tucek, J., Mancini, D.C., Moldovan, N., Erdemir, A., Ersoy, D., Gardos, M.N., Busmann, H.G., Meyer, E.M., Ding, M.Q., "Ultrananocrystalline diamond thin films for MEMS and moving mechanical assembly devices," *Diamond and Related Mat.* 10, 1952-1961 (2001).
- [21] Auciello, O., Suman, A.V., Goldsmith, C., O'Brien, S., Sampath, S., Gudeman, C., Wang, W., Hwang, J.C.M., Swonger, J., Carlisle, J.A., Balachandran, S. and Mancini, D.C., "Fundamentals and technology for monolithically integrated RF MEMS switches with ultra-nanocrystalline diamond dielectric/CMOS devices," *Proc. SPIE* 7679, K1-K9 (2010).
- [22] Reddyhoff, T., Ku, I.S.Y., Holmes, A.S. and Spikes, H.A., "Friction modifier behaviour in lubricated MEMS devices," *Tribology Letters* 41, 239-246 (2011).
- [23] Fan, L.-S., Tai, Y.-C. and Muller, R.S., "IC-processed electrostatic micromotors," *Sensors and Actuators* 20, 41-47 (1989).
- [24] Asay, D.B., Dugger, M.T., Ohlhausen, J.A., and Kim, S.H., "Macro- to nanoscale wear prevention via molecular adsorption," *Langmuir* 24, 155-159 (2008).
- [25] Asay, D.B., Dugger, M.T., and Kim, S.H., "In-situ vapor-phase lubrication of MEMS," *Tribology Letters* 29, 67-74 (2008).
- [26] Strawhecker, K., Asay, D.B., McKinney, J. and Kim, S.H., "Reduction of adhesion and friction of silicon oxide surface in the presence of n-propanol vapor in the gas phase," *Tribology Letters* 19, 17-21 (2005).
- [27] Graham, E.E. and Klaus, E.E., "Lubrication from the Vapor Phase at High Temperature," *ASLE Trans.* 29, 229-234 (1986).
- [28] Barnette, A.L., Asay, D.B., Ohlhausen, J.A., Dugger, M.T. and Kim S.H., "Tribochemical polymerization of adsorbed n-pentanol on SiO₂ during rubbing: when does it occur and is it responsible for effective vapor phase lubrication?," *Langmuir* 26, 16299-16304 (2010).
- [29] Kemme, H.R. and Kreps, S.I., "Vapor pressure of primary n-alkyl chlorides and alcohols," *J. Chem. Eng. Data* 14, 98-102 (1969).